

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Stoyanov et. al.

Attorney Docket No. 25384

Application No. 10/815,206

Group Art Unit: 1731

Filed: 03/31/2004

Examiner: Cordray, Dennis R.

Title: Individualized Intrafiber Crosslinked Cellulosic Fiber With Improved Brightness and Color

DECLARATION OF ANGEL STOYANOV PURSUANT TO § 37 C.F.R. § 1.132

Federal Way, WA,

October 4, 2006

TO THE COMMISSIONER OF PATENTS:

I, Angel Stoyanov, declare and state as follows:

1. I am currently employed by the Weyerhaeuser Company as a Scientist and since 1998 have worked exclusively on crosslinking of cellulosic fibers.

2. I received my Bachelor of Science and my Master Of Science from the University of Chemical Technology and Metallurgy at Sofia, Bulgaria, in 1980 and 1981, respectively. After graduation my work history is as follows:

I was a Research Assistant from 1982 to 1986 and an Assistant Professor from 1986 to 1994 at the University of Chemical Technology and Metallurgy at Sofia, Bulgaria. From 1990 to 1991 I worked under a Fulbright scholarship at the University of Washington, Seattle, WA, and completed all graduate courses for a Ph. D. in 1996. From

1996 to 1998 I conducted research for my Ph. D. and held various teaching positions in the Department of Engineering at the University of Washington.

3. I have read and am familiar with the Hansen et al patent US Patent No. 6,340,411

4. Hansen et al state in the '411 patent that initial application of the binder on high bulk fibers preferably occurs after the curing step, particularly if the binder is capable of functioning as a crosslinking material. Hansen then states that specific binders that can also crosslink are polyols, polyaldehydes, polycarboxylic acids and polyamines. If such binders are present during curing, the binder will be consumed during the curing step to form covalently crosslinked bonds. When this occurs, the binder is no longer available for hydrogen bonding or coordinate covalent bonding, and particle binding to fibers is ineffective, column 34, line 1-13.

5. Tests were undertaken to determine if polyols indeed act as crosslinking agents with cellulose. Accordingly, I planned and supervised experiments which were carried out by my technician Derik Rieger.

6. Exhibit A shows the experimental design for the tests. All samples were cured at 171°C for 7 minutes. The acronyms are as follows: COP, chemical on pulp (CF416 pulp from Weyerhaeuser Co.); SHP, sodium hypophosphite; CA, citric acid; SOR, sorbitol; and XYL, xylitol. Exhibit B shows the addition levels for the various reagents; Exhibit C gives the procedure, Exhibit D shows the results of brightness testing by TAPPI T 525 om-02 and Exhibit E, the FAQ wet bulk results determined by the procedure in the application. The Hunter color values were determined by TAPPI T 1231 sp 98. Whiteness Index, WI<sub>(CDM-L)</sub>, was calculated from the formula, WI<sub>(CDM-L)</sub> = (L-3b).

7. The results are summarized in Table 1.

Table 1

## Fiber Properties

Sample	Wt. % on Dry Fiber				ISO				Hunter Color		Wt.(CDM-L)
	CA	SHP	Sorbitol	Xylitol	FAQ Wet Bulk, cc/g	Brightness %	L	a	b		
A	0	0	0	0	11.59	82.7	94.9	-0.83	5.58	78.16	
B	0	2	0	0	12.26	82.8	95.0	-0.83	5.58	77.87	
C	8	2	0	0	18.48	78.5	94.7	-2.02	8.67	68.69	
D	8	2	2	0	18.29	83.7	95.3	-1.41	5.53	78.71	
E	8	2	6	0	17.05	85.4	95.7	-1.23	4.80	81.3	
F	8	2	0	2	18.18	84	95.6	-1.45	5.7	78.50	
G	8	2	0	6	16.83	85.8	95.7	-1.21	4.53	82.10	
H	0	2	2	0	11.43	82.3	94.8	-0.88	5.81	77.37	
I	0	2	6	0	11.10	81.4	94.4	-0.81	5.96	76.52	
J	0	2	0	2	11.27	80.5	94.1	-0.78	6.20	75.50	
K	0	2	0	6	10.76	79.8	93.3	-0.76	5.60	76.50	

8. It is well recognized by those skilled in the art of crosslinked fibers that an increase in FAQ wet bulk, relative to an untreated control, reflects that fibers have been crosslinked.

9. Sample A is a control and Sample B is the pulp with 2 percent by dry weight sodium hypophosphate; FAQ wet bulk values are 11.59 and 12.26 cc/g, respectively, and  $WI_{(CDM-L)}$  values are 78.16 and 77.87, respectively. When pulp is treated with citric acid and sodium hypophosphate, Sample C, FAQ wet bulk is 18.48 cc/g and the Whiteness Index is 68.69. When pulp is treated with citric acid, sodium hypophosphate and sorbitol, a polyol, at the 2 and 6 percent by weight level of sorbitol on pulp, Samples D and E, respectively, FAQ wet bulk is significantly increased to 18.29 and 17.05 cc/g, respectively. The Whiteness Index of Samples D and E, also increased to 78.71 and 81.30, respectively. However, when pulp is treated only with sodium hypophosphate and two different levels of sorbitol, 2 and 6 percent by weight, Samples H and I, there is no increase in FAQ wet bulk; Whiteness Index, decreased relative to the control pulp and the pulp sample with only sodium hypophosphate, Samples A and B, respectively.

When pulp is treated with citric acid, sodium hypophosphate and xylitol, a polyol, at the 2 and 6 percent by weight level of xylitol on pulp, Samples F and G, respectively, FAQ wet bulk is significantly increased to 18.18 and 16.83 cc/g, respectively. The Whiteness Index of Samples F and G, also increased to 78.50 and 82.10, respectively. However, when pulp is treated only with sodium hypophosphate and two different levels of xylitol, 2 and 6 percent by weight, Samples J and K, there is no increase in FAQ wet bulk; Whiteness Index  $WI_{(CDM-L)}$ , decreased relative to the control pulp and the pulp with only sodium hypophosphate, Samples A and B, respectively.

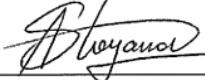
10. Based on the fact that there is no increase in FAQ wet bulk when pulp is treated only with sodium hypophosphate and sorbitol, or only with sodium hypophosphate and xylitol, it is my opinion that the polyol, sorbitol, and the polyol, xylitol, do not crosslink with cellulose.

11. In accordance with accepted Patent Office Practice, the dates in the laboratory notebook pages presented in Exhibits A- E have been redacted.

12. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Respectfully submitted,

Date 10/4/06



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Angel Stoyanov

## EXHIBIT A

84

Project No.  
Book No. 14680TITLE Expt 145 Solutions

From Page No.

Weyerhaeuser Confidential

Patent Action

Due Date: 8/4/2006

Title: Experiment # 145: CA + Polyols for Patent action

Objective(s): Investigate whether polyols will be involved in crosslinking of cellulose fibers under the conditions used for esterification of cellulose with CA

Materials:

- Pulp: CF416 - 94%
- Sample size: 20 g
- Xlinker: CA
- Catalyst: SHP <sup>99</sup>
- Polyols: Sorbitol (Sorbitex) and Xylitol (Xylidex) <sup>98%</sup>
- Fiberizer: 6" pad former
- Despatch oven
- Metal baskets for curing

Experimental Design:

Sample ID	Chemistry	XLinker	SHP (% COP)	Polyol		Cure Temp. (°F)	Cure time (min.)
				Sorbitol (% COP)	Xylitol (% COP)		
A	Blank		0	0	0	340	7
B	Pulp+SHP		0	2	0	340	7
C	CA+SHP		8	2	0	340	7
D	CA+SHP+SOR		8	2	2	340	7
E	CA+SHP+SOR		8	2	6	340	7
F	CA+SHP+XYL		8	2	0	340	7
G	CA+SHP+XYL		8	2	0	340	7
H	SHP+SOR		0	2	2	340	7
I	SHP+SOR		0	2	6	340	7
J	SHP+XYL		0	2	0	340	7
K	SHP+XYL		0	2	0	340	7

Procedure:

1. Weigh the sample 20 g (odb);
2. Apply the crosslinking solution using the usual syringe method;
3. Leave the samples overnight in a sealed plastic bags;
4. Use the 6" pad former for fluffing (50% consistency);
5. Cure the samples in the Despatch V Series oven;
6. Store the cured fibers in a plastic bag.

Testing:

1. AFAQ Wet Bulk at 0.6 kPa
2. Brightness/Color

Witnessed &amp; Understood by me,

Date

Invented by

Recorded by

Date

8/1/06

## EXHIBIT B

Project No. 14680  
Book No. 14680

85

TITLE Expt 145 SolutionsFrom Page No. 1

## Exp# 145:CA+ Polyols for patent action

Date: 8/1/2006

Pulp CF416

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
A	CA	0	20	100	0.000	
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	0	20	1.20	0.000	

pH 7.15

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
B	CA	0	20	100	0.000	
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.483

pH 7.06

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
C	CA	8	20	100	1.600	1.597
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.482

pH 7.96

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
D	CA	8	20	100	1.600	1.603
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.479
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Sorbitol	2	20	100	0.400	0.401

pH 7.91

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
E	CA	8	20	100	1.600	1.603
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.480
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Sorbitol	6	20	100	1.200	1.202

pH 7.93

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
F	CA	8	20	100	1.600	1.605
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.480
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Xyitol	2	20	100	0.400	0.400

pH 7.92

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
G	CA	8	20	100	1.600	1.601
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.481
	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
	Xyitol	6	20	100	1.200	1.191

pH 7.94

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
H	Sorbitol	2	20	100	0.400	0.399
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.485

pH 7.73To Page No. 1

Witnessed &amp; Understood by me,

Date

Invented by Steve D. Pogler

Date

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## EXHIBIT B

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Project No.  
Book No. 14680

TITLE 894 14S Solutions NATA

From Page No. --

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
I	Sorbitol	6	20	100	1.200	1.202
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.482

pH 4.72

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
J	Xylitol	2	20	100	0.400	0.401
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.489

pH 4.75

Sample ID	Reagent	%Concentration	Final Volume(g)	%Solids	Amount to be weighed	Actual amount
K	Xylitol	6	20	100	1.200	1.199
	Reagent	%Concentration	Final Volume(g)	SHP formula	Amount to be weighed	Actual amount
	SHP	2	20	1.20	0.482	0.484

pH 4.75

To Page No. --

Witnessed &amp; Understood by me,

Date

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Recorded by

Date

8/10/06

## EXHIBIT C

TITLE Expt #145 : CA + Polyols for Patent adu Project No. 14500 Book No. 14500

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From Page No.

8/1/06

	<u>Target weight (g)</u>	<u>Actual weight (g)</u>	<u>Pulp + solution (g)</u>
A)	21.20	21.29	41.88
B)		21.15	41.06
C)		21.21	41.15
D)		21.38	41.26
E)		21.34	41.31
F)		21.23	41.33
G)		21.37	41.17
H)		21.15	41.03
I)		21.84	41.13
J)		21.25	40.98
K)		21.22	41.13

- Prepared solutions on 8/1/06
  - Applied to sheets
- Fiberized on 8/2/06 - Visual on fibres appears to be no different between samples, pre-curing.
- Samples air dry on table top for 4 hours before curing.
- Samples cured @ 340° for 7 min each on 8/2/06
- Samples placed in 50% humidity room before FAQ testing, 8/3/06
- TESTED: Brightness + color on 8/3/06
- FAQ TESTER in MIZZUNE not used after many controls would not come into spec. ON 8/3/06
  - 8/3/06 FAQ Tester in 116 was used

Witnessed &amp; Understood by me,

Date

Invented by

Eric Fletcher

Recorded by

To Page No.

Date

8/4/06

## EXHIBIT D

88

Project No.  
Book No. 14680

TITLE EXHIT 145

Brightness Results

From Page No.

Exp#	Sample#	side	position	Operator	TEST DATE	BRIGHTNESS	R(X)	R(Y)	R(Z)	X	Y	Z	L	a	
145	A	a	1	D	08/03/06	82.44	91.01	89.69	82.21	87.52	89.89	97.2	94.7	-0.84	
145	A	a	2		08/03/06	82.42	88.65	82.18	87.5	89.85	97.16	94.69	-0.8		
145	A	a	3		08/03/06	82.39	90.98	88.44	82.14	87.48	89.64	97.12	94.68	-0.92	
145	A	b	1		08/03/06	83.03	91.79	80.41	82.78	88.24	90.41	97.85	95.68	-0.81	
145	A	b	2		08/03/06	83.05	91.8	80.44	82.79	88.25	90.44	97.88	95.1	-0.85	
145	A	b	3		08/03/06	83.04	91.77	80.42	82.78	88.23	90.42	97.87	95.09	-0.85	
					Average	82.7	91.4	89.0	82.5	87.9	89.8	97.5	94.8	-0.8	
					StDev	0.3	0.4	0.4	0.3	0.4	0.4	0.4	0.2	0.0	
145	B	a	1	D	08/03/06	81.85	91.13	89.58	81.58	87.49	89.58	96.45	94.7	-0.88	
145	B	a	2		08/03/06	81.87	91.05	89.58	81.42	87.39	89.58	96.27	94.85	-0.87	
145	B	a	3		08/03/06	81.87	91.07	89.59	81.4	87.41	89.59	96.24	94.85	-0.87	
145	B	b	1		08/03/06	83.8	92.16	90.92	83.57	88.69	90.92	98.81	95.85	-0.91	
145	B	b	2		08/03/06	83.82	92.2	90.94	83.57	88.72	90.94	98.81	95.38	-0.86	
145	B	b	3		08/03/06	83.79	92.15	90.89	83.55	88.68	90.89	98.79	95.34	-0.87	
					Average	82.8	91.6	90.3	82.5	88.1	90.3	97.6	95.0	-0.9	
					StDev	1.1	0.6	0.7	1.2	0.7	0.7	1.4	0.4	0.0	
145	C	a	1	D	08/03/06	78.52	91.12	69.54	77.97	86.77	80.54	92.19	94.53	-1.98	
145	C	a	2		08/03/06	78.54	91.12	69.58	77.98	86.77	80.55	92.2	94.63	-2	
145	C	a	3		08/03/06	78.58	91.19	69.83	78.02	86.83	80.63	92.25	94.87	-2.02	
145	C	b	1		08/03/06	78.29	91.2	69.59	77.72	86.79	80.59	91.85	94.85	-2.03	
145	C	b	2		08/03/06	78.61	91.57	69.93	78.02	87.13	80.93	92.24	94.83	-2.02	
145	C	b	3		08/03/06	78.07	91.53	69.92	78.07	87.11	80.92	92.31	94.83	-2.04	
					Average	78.5	91.3	69.7	78.0	86.9	80.7	92.2	94.7	-2.0	
					StDev	0.1	0.2	0.2	0.1	0.2	0.2	0.1	0.1	0.0	
145	D	a	1	D	08/03/06	83.84	91.07	81.05	83.47	85.02	91.05	88.65	95.62	-1.46	
145	D	a	2		08/03/06	84.11	91.22	81.28	83.74	85.11	91.22	88.65	95.24	-1.48	
145	D	a	3		08/03/06	84.05	92.33	91.37	83.88	88.68	91.37	95.59	93.59	-1.38	
145	D	b	1		08/03/06	83.29	91.33	90.28	82.88	87.9	90.38	98	95.07	-1.38	
145	D	b	2		08/03/06	83.35	91.41	90.45	82.94	87.98	90.45	98.06	95.1	-1.37	
145	D	b	3		08/03/06	83.5	91.52	90.59	83.09	88.09	90.59	98.24	95.16	-1.42	
					Average	83.7	91.6	89.9	83.3	88.4	90.9	98.5	95.3	-1.4	
					StDev	0.4	0.4	0.4	0.4	0.4	0.4	0.5	0.2	0.0	
145	E	a	1	D	08/03/06	85.07	92.18	91.39	84.78	88.94	91.39	100.23	95.6	-1.28	
145	E	a	2		08/03/06	85.52	92.57	91.75	85.19	89.33	91.75	100.72	95.78	-1.22	
145	E	a	3		08/03/06	85.63	92.63	91.8	85.22	89.39	91.8	100.81	95.81	-1.19	
145	E	b	1		08/03/06	85.11	92.16	91.37	84.81	89.63	91.37	100.27	95.59	-1.26	
145	E	b	2		08/03/06	85.34	92.42	91.6	85	89.17	91.6	100.5	95.71	-1.23	
145	E	b	3		08/03/06	85.7	92.89	91.88	85.36	89.48	91.88	100.92	95.86	-1.22	
					Average	85.4	92.4	91.6	85.1	89.2	91.6	100.6	95.7	-1.2	
					StDev	0.3	0.2	0.2	0.2	0.2	0.2	0.3	0.1	0.0	
145	F	a	1	D	08/03/06	83.8	92.07	91.04	83.22	88.55	91.08	88.30	95.44	-1.45	
145	F	a	2		08/03/06	83.91	92.35	91.34	83.48	88.62	91.34	88.71	95.57	-1.43	
145	F	a	3		08/03/06	83.94	92.38	91.39	83.49	88.65	91.35	88.71	95.5	-1.46	
145	F	b	1		08/03/06	83.09	92.24	91.3	83.2	88.76	91.3	88.65	95.55	-1.47	
145	F	b	2		08/03/06	84.17	92.4	91.43	83.73	88.81	91.43	99	95.62	-1.43	
145	F	b	3		08/03/06	84.09	92.31	91.38	83.69	88.63	91.38	95.05	95.86	-1.48	
					Average	84.9	92.3	91.3	83.5	88.8	91.3	98.8	95.6	-1.5	
					StDev	0.2	0.1	0.1	0.2	0.1	0.1	0.2	0.1	0.0	
145	G	a	1	D	08/03/06	85.7	92.25	91.54	85.21	89.17	91.54	100.02	95.68	-1.23	
145	G	a	2		08/03/06	85.09	92.65	91.9	85.74	89.5	91.8	101.35	95.38	-1.17	
145	G	a	3		08/03/06	86.04	92.67	91.89	86.74	89.51	91.99	101.37	95.86	-1.14	
145	G	b	1		08/03/06	85.85	92.46	91.71	85.53	89.31	91.71	101.13	95.77	-1.19	
145	G	b	2		08/03/06	85.86	92.31	91.55	85.29	89.14	91.55	100.85	95.65	-1.21	
145	G	b	3		08/03/06	85.47	92.13	91.43	85.16	88.88	91.43	100.69	95.62	-1.3	
					Average	85.8	92.4	91.7	85.5	89.3	91.7	101.1	95.7	-1.2	
					StDev	0.2	0.2	0.2	0.2	0.2	0.2	0.3	0.1	0.1	
145	H	a	1	D	08/03/06	82.22	91.17	89.73	81.93	87.59	89.73	98.87	94.75	-0.88	
145	H	a	2		08/03/06	82.22	91.15	89.75	81.93	87.57	89.75	98.87	94.74	-0.85	
145	H	a	3		08/03/06	82.17	91.09	89.72	81.87	87.51	89.72	97.99	94.72	-0.9	
145	H	b	1		08/03/06	82.43	91.36	89.97	82.12	87.78	89.97	97.09	94.85	-0.88	
145	H	b	2		08/03/06	82.35	91.31	89.93	82.05	87.72	89.93	97.01	94.83	-0.89	
145	H	b	3		08/03/06	82.26	91.28	89.9	82.07	87.60	89.9	95.01	94.81	-0.89	
					Average	82.3	91.2	89.8	82.0	87.8	89.8	98.9	94.8	-0.8	
					StDev	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0	

Signature

To Page No.

-Witnessed &amp; Understood by me, -

Date

Invented by

Suzanne D. Green

Date

8/10/06

#### EXHIBIT D

TITLE Exhibit 145 Brightness Results

Book No. 14/080

39

From Page No.

Re: 2/20/06

HUNTER W3TM/CIE W CIE TINT

	I <sup>+</sup>	I <sup>-</sup>	J <sup>+</sup>	J <sup>-</sup>	B <sup>+</sup>	B <sup>-</sup>		HUNTER WSTMICRO W. CHE TINT
5.52	95.87	-0.81	5.51	0	0	575.06	5.25	58.89 59.8 64.42 -1.58
5.52	95.85	-0.77	5.51	0	0	575.15	5.26	58.85 50.77 -6.35 -1.89
5.54	95.85	-0.79	5.54	0	0	575.11	5.28	58.64 58.64 64.27 -1.57
5.63	96.17	-0.78	5.62	0	0	575.16	5.34	59.41 59.81 64.74 -2.03
5.63	96.18	-0.82	5.62	0	0	575.07	5.34	59.44 59.83 64.77 -1.97
5.62	96.17	-0.82	5.61	0	0	575.05	5.33	59.42 59.82 64.79 -1.95
5.8	96.6	-0.8	5.6	0.0	0.0	575.1	5.3	59.8 59.8 -6.46 -2.0
0.1	0.2	0.0	0.1	0.0	0.0	0.0	0.4	0.1 0.2 0.0
5.89	95.87	-0.85	5.89	0	0	575.12	5.71	58.68 57.27 62.21 -2.14
6.03	95.82	-0.84	6.04	0	0	575.17	5.76	58.95 50.96 61.89 -2.19
6.08	95.83	-0.84	6.06	0	0	575.18	5.79	59.59 56.83 61.77 -2.21
5.4	96.38	-0.87	5.37	0	0	574.8	5.08	59.92 61.51 68.42 -1.72
5.41	95.39	-0.85	5.38	0	0	574.87	5.09	59.04 61.46 68.39 -1.76
5.39	96.37	-0.84	5.38	0	0	574.89	5.07	59.89 61.53 68.43 -1.77
5.7	96.1	-0.8	5.7	0.0	0.0	575.0	5.4	59.63 59.3 64.2 -2.0
0.3	0.3	0.0	0.4	0.0	0.0	0.2	0.4	0.7 2.5 2.4 0.2
8.56	95.81	-1.92	8.69	0	0	573.8	8.17	59.54 43.27 49.55 -1.85
8.58	95.81	-1.94	8.69	0	0	573.75	8.17	59.56 43.27 49.55 -1.85
8.58	95.84	-1.96	8.71	0	0	573.72	8.19	59.63 43.2 49.57 -1.78
8.78	95.82	-1.97	8.92	0	0	573.79	8.39	59.59 42.12 48.57 -1.85
8.79	95.97	-1.98	8.93	0	0	573.82	8.39	59.93 42.28 48.61 -1.91
8.75	95.88	-1.97	8.88	0	0	573.77	8.34	59.82 42.53 49.12 -1.85
8.7	95.9	-2.0	8.8	0.0	0.0	573.8	8.17	59.7 42.8 42.8 40.2 -1.8
0.1	0.4	0.1	0.0	0.0	0.0	0.1	0.2	0.5 0.4 0.4
5.58	96.43	-1.41	5.54	0	0	573.29	5.14	61.05 80.73 65.77 -0.85
5.55	96.82	-1.41	5.52	0	0	573.28	5.12	61.26 60.97 69.09 -0.86
5.5	96.56	-1.33	5.47	0	0	573.49	5.08	61.37 61.32 66.44 -0.97
5.52	96.15	-1.33	5.5	0	0	573.5	5.12	59.38 60.41 65.23 -0.98
5.52	96.18	-1.32	5.51	0	0	573.54	5.13	59.45 60.43 65.27 -1.01
5.51	96.24	-1.38	5.49	0	0	573.37	5.11	59.59 60.6 65.49 -0.9
5.5	96.3	-1.4	5.5	0.0	0.0	573.4	5.1	59.9 60.7 65.7 -0.9
0.0	0.2	0.0	0.0	0.0	0.0	0.1	0.0	0.4 0.5 0.1
4.84	96.57	-1.24	4.8	0	0	573.26	4.44	61.39 64.95 66.55 -0.73
4.79	95.72	-1.17	4.75	0	0	573.43	4.4	61.75 65.51 70.16 -0.81
4.78	95.74	-1.15	4.73	0	0	573.51	4.38	61.98 65.65 70.3 -0.84
4.8	95.56	-1.22	4.78	0	0	573.29	4.4	61.37 65.14 69.7 -0.74
4.82	95.65	-1.19	4.78	0	0	573.41	4.42	61.96 65.22 69.66 -0.8
4.75	95.77	-1.18	4.71	0	0	573.39	4.36	61.68 65.79 70.45 -0.78
4.8	96.7	-1.2	4.8	0.0	0.0	573.4	4.4	61.6 65.4 70.0 -0.8
0.0	0.1	0.0	0.0	0.0	0.0	0.1	0.0	0.4 0.5 0.1
5.77	95.81	-1.38	5.75	0	0	573.24	5.31	61.03 59.84 64.66 -1
5.75	95.65	-1.38	5.73	0	0	573.52	5.33	61.34 59.91 65.21 -1.03
5.78	95.57	-1.41	5.76	0	0	573.48	5.35	61.39 59.79 65.13 -1
5.64	95.53	-1.42	5.61	0	0	573.33	5.2	61.93 60.51 65.71 -0.9
5.64	95.59	-1.38	5.61	0	0	573.44	5.21	61.43 60.83 65.67 -0.96
5.63	95.67	-1.43	5.61	0	0	573.3	5.19	61.38 60.62 65.83 -0.88
5.7	96.5	-1.4	5.7	0.0	0.0	573.4	5.3	61.93 62.2 65.4 -1.0
0.1	0.1	0.0	0.1	0.0	0.0	0.1	0.1	0.5 0.4 0.1
5.77	95.81	-1.38	5.75	0	0	573.24	5.31	61.03 59.84 64.66 -1
5.75	95.65	-1.38	5.73	0	0	573.52	5.33	61.34 59.91 65.21 -1.03
5.78	95.57	-1.41	5.76	0	0	573.48	5.35	61.39 59.79 65.13 -1
5.64	95.53	-1.42	5.61	0	0	573.33	5.2	61.93 60.51 65.71 -0.9
5.64	95.59	-1.38	5.61	0	0	573.44	5.21	61.43 60.83 65.67 -0.96
5.63	95.67	-1.43	5.61	0	0	573.3	5.19	61.38 60.62 65.83 -0.88
5.7	96.5	-1.4	5.7	0.0	0.0	573.4	5.3	61.93 62.2 65.4 -1.0
0.1	0.1	0.0	0.1	0.0	0.0	0.1	0.1	0.5 0.4 0.1
4.52	96.83	-1.18	4.47	0	0	573.17	4.13	61.54 68.81 71.18 -0.64
4.49	96.78	-1.12	4.44	0	0	573.35	4.11	61.9 67.28 71.72 -0.72
4.49	95.78	-1.1	4.44	0	0	573.45	4.11	61.88 67.28 71.71 -0.76
4.52	96.7	-1.14	4.47	0	0	573.31	4.13	61.71 67 71.41 -0.7
4.57	95.83	-1.17	4.52	0	0	573.26	4.18	61.55 68.54 70.96 -0.69
4.59	95.69	-1.25	4.54	0	0	573.67	4.19	61.43 68.35 70.75 -0.55
4.5	95.87	-1.2	4.5	0.0	0.0	573.3	4.1	61.7 66.9 71.3 -0.7
0.1	0.1	0.1	0.0	0.0	0.0	0.2	0.0	0.4 0.4 0.1
5.8	95.01	-0.84	5.8	0	0	575.06	5.52	58.78 58.37 63.21 -2.03
5.78	95.69	-0.82	5.77	0	0	575.12	5.5	59.75 58.49 63.3 -2.06
5.8	95.68	-0.87	5.8	0	0	574.99	5.52	58.92 58.32 63.14 -1.99
5.8	95.68	-0.84	5.79	0	0	575.07	5.51	58.97 58.55 63.45 -2.03
5.82	95.97	-0.88	5.81	0	0	575.02	5.53	58.93 58.41 63.31 -2.01
5.85	95.95	-0.88	5.85	0	0	575.04	5.57	58.9 58.18 63.1 -2.04
5.8	95.9	-0.8	5.8	0.0	0.0	575.1	5.5	58.98 58.4 63.3 -2.0

To Page No.

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Date

81.10106

## EXHIBIT D

90

Project No.  
Book No. 14660TITLE Expt 145 Brightness Results

From Page No. 1

Expt	Sample#	side	position	Operator	TEST DATE	BRIGHTNESS	R(X)	R(Y)	R(Z)	X	Y	Z	L	a
145	I	a	1	D	08/03/06	81.45	90.64	89.16	81.19	87.03	89.16	96	94.43	-0.6
145	I	a	2		08/03/06	81.47	90.63	89.16	81.21	87.03	89.16	96.01	94.42	-0.79
145	I	a	3		08/03/06	81.26	90.62	89.09	81.11	86.95	89.09	95.59	94.39	-0.81
145	I	b	1		08/03/06	81.46	90.77	89.27	81.16	87.13	89.27	95.98	94.46	-0.81
145	I	b	2		08/03/06	81.39	90.71	89.21	81.07	87.09	89.21	95.85	94.45	-0.83
145	I	b	3		08/03/06	81.36	90.73	89.21	81.08	87.08	89.21	95.88	94.45	-0.79
					Average	81.4	90.7	89.2	81.1	87.0	89.2	95.9	94.4	-0.6
					StdDev	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.0	0.0
145	J	a	1	D	08/03/06	80.46	90.08	88.51	80.1	86.39	85.51	94.51	94.08	-0.78
145	J	a	2		08/03/06	80.47	90.05	88.47	80.2	86.37	85.47	94.42	94.06	-0.77
145	J	a	3		08/03/06	80.33	89.95	88.38	80.07	86.27	85.38	94.57	94.01	-0.78
145	J	b	1		08/03/06	80.72	90.36	88.76	80.45	86.96	85.79	95.12	94.22	-0.78
145	J	b	2		08/03/06	80.59	90.27	88.65	80.3	86.57	86.68	94.04	94.17	-0.77
145	J	b	3		08/03/06	80.48	90.19	88.6	80.2	86.48	86.5	94.82	94.13	-0.79
					Average	80.5	90.2	88.6	80.2	86.5	85.8	94.9	94.1	-0.6
					StdDev	0.1	0.2	0.1	0.1	0.1	0.2	0.1	0.0	0.0
145	K	a	1	D	08/03/06	80.24	88.94	87.59	80	85.46	87.58	94.59	93.58	-0.6
145	K	a	2		08/03/06	80.3	88.99	87.59	80.06	85.51	87.59	94.65	93.59	-0.75
145	K	a	3		08/03/06	80.29	88.97	87.57	80.05	85.5	87.57	94.64	93.55	-0.74
145	K	b	1		08/03/06	79.49	87.99	86.63	79.25	84.57	86.63	93.7	93.07	-0.76
145	K	b	2		08/03/06	79.35	87.67	86.5	79.1	84.45	86.5	93.62	93	-0.75
145	K	b	3		08/03/06	79.33	87.86	86.5	79.08	84.43	86.5	93.5	93.01	-0.78
					Average	79.8	88.4	87.1	79.6	85.0	87.1	94.1	93.3	-0.6
					StdDev	0.5	0.6	0.6	0.5	0.6	0.6	0.6	0.3	0.0

*devalue*

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Invented by

*Beck Ridge*

Recorded by

To Page No.

## EXHIBIT D

TITLE ENQ # 145 Brightness Results Book No. 14680

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From Page No. 1*ok-31-81*

b	L*	a*	b*			HUNTER W5TM/CIE V. CIE TINT					
5.01	95.65	-0.77	5.91	0	575.32	5.66	89.16	57.29	62	-2.25	
5.02	95.65	-0.76	5.9	0	575.34	5.65	89.16	57.38	62.05	-2.26	
5.02	95.62	-0.78	5.93	0	575.27	5.68	89.09	57.16	81.85	-2.22	
5	95.69	-0.78	6.01	0	575.3	5.75	89.27	50.88	61.69	-2.27	
6.04	95.67	-0.8	6.05	0	575.27	5.79	89.21	56.63	61.44	-2.27	
6.02	95.67	-0.76	6.03	0	575.38	5.78	89.21	56.7	61.5	-2.33	
6.0	95.7	-0.8	6.0	0.0	575.3	5.7	89.2	57.6	61.8	-2.3	
0.1	0.0	0.0	0.1	0.0	0.0	0.1	0.1	0.3	0.3	0.0	
6.19	95.37	-0.78	6.21	0	575.44	5.97	88.51	55.25	59.9	-2.45	
6.10	95.38	-0.74	6.18	0	575.48	5.95	88.47	55.38	60	-2.47	
6.19	95.32	-0.75	6.21	0	575.46	5.98	88.38	55.14	58.74	-2.47	
6.10	95.49	-0.75	6.21	0	575.46	5.97	88.78	55.46	60.21	-2.48	
6.23	95.45	-0.75	6.25	0	575.48	6.01	88.68	55.16	59.9	-2.49	
6.25	95.41	-0.76	6.27	0	575.44	6.03	88.6	55	59.72	-2.48	
6.2	95.4	-0.8	6.2	0.0	575.5	6.0	88.6	55.2	59.9	-2.5	
0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.1	0.2	0.2	0.0	
5.08	94.98	-0.77	5.67	0	575.22	5.48	87.56	57.32	61.34	-2.1	
5.03	94.09	-0.73	5.65	0	575.34	5.44	87.59	57.46	61.48	-2.17	
5.63	94.98	-0.71	5.65	0	575.37	5.44	87.57	57.47	61.49	-2.19	
5.54	94.58	-0.73	5.57	0	575.29	5.38	86.63	57.14	60.8	-2.12	
5.57	94.53	-0.72	5.6	0	575.33	5.41	86.5	56.9	60.53	-2.15	
5.58	94.53	-0.76	5.61	0	575.23	5.43	86.5	56.61	60.46	-2.1	
5.6	94.8	-0.7	5.6	0.0	575.3	5.4	87.1	57.2	61.0	-2.1	
0.0	0.2	0.0	0.0	0.0	0.0	0.1	0.0	0.6	0.3	0.5	0.0

To Page No. 1

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Invented by

Recorded by

Date

*John F. Fager*  
S. J. White

## EXHIBIT E

Project No. \_\_\_\_\_  
Book No. 16(68)

TITLE. Expt 148 FAZ Results

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From Page No.

Test	Ref # or Code	Sample	Imp	Frontizer	Fluff	Manufac	Manufac	Operator	Lab	Run	Dry Bulks	Dry Bulks	Wt%	Wt%	Wt Bulks	Wt Bulks	Wt%
		Number	Grade	Date	Type	Initial	Initial	Name	Number	code	code	code	code	mm/s	code	code	code
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	40.84	23.39	2.6	8.65	3.98	11.74	12.32	12.32
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	34.57	23.17	2.4	10.05	3.63	11.49	11.22	11.22
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	40.82	24.32	2.5	10.62	3.75	11.95	12.08	12.08
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	40.11	23.96	2.6	10.27	3.78	11.95	11.9	11.9
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	40.84	23.39	2.6	8.65	3.98	11.74	12.32	12.32
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	40.24	24.32	2.1	12.03	15.52	12.26	12.62	12.62
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	40.68	24.28	2.6	10.4	15.46	12.32	12.55	12.55
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	40.12	24.28	2.6	10.31	15.53	12.26	12.54	12.54
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	41.16	23.29	2.8	10.33	15.49	12.34	12.49	12.49
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	44.02	26.38	3	10.95	15.79	18.74	18.65	18.65
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	42.03	25.61	3.2	10.03	15.58	18.55	18.82	18.82
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	41.25	25.79	3.1	10.25	15.62	18.65	18.59	18.59
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	43.7	26.16	3.1	10.48	15.53	18.48	18.68	18.68
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	44.07	26.76	3.3	10	15.59	18.48	18.53	18.53
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	41.38	25.99	3.2	10.27	14.97	17.78	18.0	18.0
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	40.38	26.08	3.1	10.42	15.34	18.48	18.53	18.53
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	41.85	25.22	2.4	12.67	13.0	16.75	16.8	16.8
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	42.36	25.61	2.8	11.13	14.38	17.28	17.01	17.01
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	42.36	25.33	2.9	10.31	14.25	17.01	17.37	17.37
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	42.27	25.39	2.7	11.48	14.14	17.03	17.26	17.26
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	44.41	26.57	3.2	10.27	15.59	18.42	18.57	18.57
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	42.34	25.34	3.1	10.22	15.22	18.41	18.59	18.59
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	42.08	25.8	2.9	10.98	15.28	18.51	18.42	18.42
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	43.06	25.97	3	10.72	15.23	18.18	18.39	18.39
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	41.7	24.96	2.8	11.8	13.79	18.0	18.9	18.9
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	44.16	25.67	2.8	11.54	14.31	17.47	17.17	17.17
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	43.06	25.48	2.7	11.43	14.12	16.04	18.05	18.05
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	42.97	26.28	2.0	11.84	14.08	18.03	18.93	18.93
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	38.31	23.41	2.8	11.04	14.06	17.01	17.59	17.59
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	41.33	23.06	2.3	11.41	10.21	11.03	11.92	11.92
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	42.1	24.28	2.1	12.69	9.96	11.49	12.06	12.06
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	41.92	24.24	2.1	12.64	9.96	11.49	12.04	12.04
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	42.8	24.51	2.4	11.13	9.75	11.17	11.39	11.39
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	42.1	24.39	2.5	10.24	9.37	10.91	11.36	11.36
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	42.81	24.51	2.5	10.27	9.99	11.22	11.58	11.58
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	42.8	24.37	2.0	10.41	9.8	11.1	11.43	11.43
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	41.78	23.42	2.1	12.17	9.37	10.91	11.49	11.49
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	42.47	23.84	2.1	11.08	10.21	11.58	11.94	11.94
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	42.47	23.58	2.3	11.49	9.88	11.27	11.71	11.71
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	42.63	23.98	2.3	11.46	9.75	11.27	11.72	11.72
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	1	42.05	23.84	2.2	11.25	9.5	11.04	11.33	11.33
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	2	42.05	23.07	2.3	11.25	9.82	10.96	10.03	10.03
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	3	41.81	23.02	2.3	11.13	9.43	10.96	11.14	11.14
1	04/20/06	exp#145	AB/Blank	0/1/2006	o	A	A	Lab 116	AV	41.81	22.93	2.3	11.42	9.28	10.76	11.18	11.18

S. Sluck

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Witnessed &amp; Understood by me,

Date

Inventor by  
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Recorded by  
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8/10/06

Date